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Statistical analysis of species interaction and optimization of copper flotation process

Studies have been carried out for analyzing the flotation performance of chalcopyrite ore from Rakha mines, Jharkhand. The full factorial methodology has been used for design of experiments. The reagents (collector and frother) dosages and time were independent variables and recovery and grade has been considered as responses. The minimum and maximum values of operating conditions were chosen after performing preliminary experiments at different operating conditions. The effect of the variables (species interaction - main and interactional) on recovery and grade were studied. In this work, the experimental data were compared to data obtained from the factorial design of experiments to check the validation of model. The results obtained for bulk were analyzed to calculate the optimized condition for effective recovery and grade.

Keywords: *Chalcopyrite ore, flotation, reagent, metallurgical performance, statistical analysis.*

Introduction

Flotation is one of the most complex mineral processing operations as it is affected by a very large number of factors. Many of these are beyond the control of mineral engineer and some cannot be even measured quantitatively. The relations between measured and controlled variables are intricately allied.

Froth flotation is the most applied beneficiation process for copper minerals, since 1905. The flotation behaviour of copper sulphide minerals was investigated by many authors (Hangone et al., 2005; Twidle et al., 1984, Owusu et al.,2015). Studies on flotation of copper ores are mainly directed towards understanding the flotation response of the ore under variable operating conditions. From literature, it has been observed that the reagent dosages (especially collector and frother dosage) and their interaction with particle size are the most critical parameters, which control the efficiency of the process. The performance of the process is mainly assessed through recovery and grade of concentrate and the overall yield, as a function of time and reagent dosages.

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Various studies have been carried out on flotation with respect to the influence of reagent quantity. The required extent of hydrophobicity, which is obtained by addition of collector, is very important as it should be adequate enough to form a monolayer on the surface of particle. A higher concentration reduces the effect of collector on the valuable minerals (Wills' Mineral Processing Technology, 2016). The relationship between collector dosage and contact angle of the mineral particle was studied by Sutherland and Wark who concluded that greater the collector concentration is more rapid will be the development of the contact angle (Sutherland et al., 1955).

Influence of the reagents on the total recovery of the floatable component and the rate at which they report to the concentrate, has been a major aspect of analysis of the flotation process. Throne et al. (1976) concluded that concentration of collector in solution determines the time taken to develop the maximum contact angle for a particular mineral surface. In the present study, the effect of reagents on flotation of chalcopyrite ore has been studied using statistical design of experiments. The response to flotation has been analysed by full factorial method. The results were used to understand the metallurgical performance of bulk flotation as a function of reagent dosages and flotation time.

Experimental design

The factorial design test is one of the most effective technique to study the process behaviour with analysis of variance (ANOVA) (Naik et al., 2002a; Naik et al., 2002b). The statistical design of experiments and analysis has been used to study the flotation behaviour of minerals (Çilek et al., 2003; Martýinez et al., 2003; Rao et al., 2002) and coal (Mohanty et al., 1999; Rao et al.,1982). Naik et al. (2004) performed a 2^5 factorial design on low rank coal flotation to study the effect of diesel oil, Methyl isobutyl Carbinol (MIBC), particle size, p^H and sodium silicate. They also used 2^3 factorial design experiments for higher rank coal flotation to study the influence of kerosene, MIBC and sodium metasilicate (Naik et al., 2005). Dariuh applied Plackett-Burman methodology for optimizing the variables to get desired responses (minimum ash content and maximum efficiency index) in coal flotation (Azizi et al., 2014).

In the present study, experimental design, analysis of results and optimization of the process were done by Design Expert 7 software. To select the range of reagent dosages at which the performance is maximum, 2^3 factorial design experiments were carried out with Sodium isopropyl xanthate (collector), pine oil (frother) and time on a low grade copper ore. The primary aim was the optimization of copper flotation condition and the effect of variables (main and interactional).

Materials and methods

Material characterization

A representative sample of copper ore was taken from the Rakha copper mines in Jharkhand, India and the entire sample was crushed to below 1mm (1000 micron). Sieving was carried out and the result of sieve analysis is presented in Fig.1. The copper content of head sample was found to be about 0.97. The size analysis showed that the d_{50} and d_{80} of the feed are 82 µm and 210 µm respectively. Since the material is finer, froth flotation was selected to upgrade the copper ore.

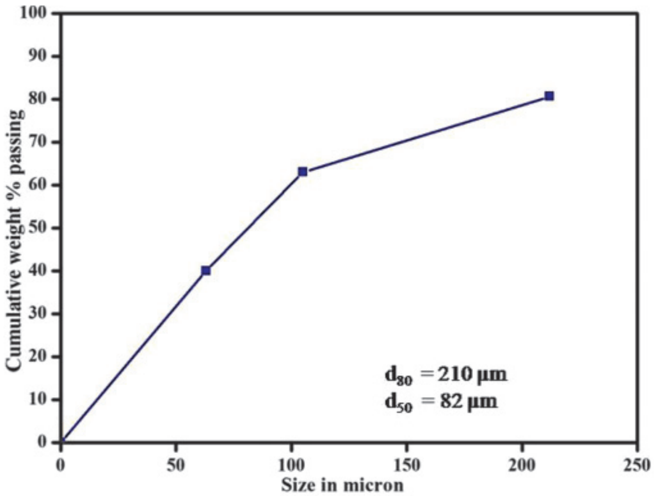

Fig.1 Size analysis of -1mm feed

Flotation experiments

Flotation experiments were carried out using an Agitair Flotation Machine of 5000ml capacity with an impeller speed of 750 rpm. 1280 g of the representative sample was used in each test to maintain the pulp density of 22%. The experimental conditions are outlined below:

- pH of slurry - 7.
- Aeration rate - 3.5 NM^3/hr.
- Conditioning time for collector - 5 minute.

Preliminary tests were done by varying collector (0.00-0.04kg/tonne) and frother (0.02-0.06 kg/tonne) dosages to find the optimum condition at which best recovery and grade was obtained. The results of the preliminary tests were used to formulate an experimental design in "Design Expert 7" software.

Results and discussion

Determination of the operating condition for flotation tests

In the initial step of investigation, collector and frother dosages and time interval were to be determined at which best results can be obtained. Dosages of collector (sodium isopropyl xanthate), frother (pine oil) and time interval were varied. The results are shown in Fig.2.

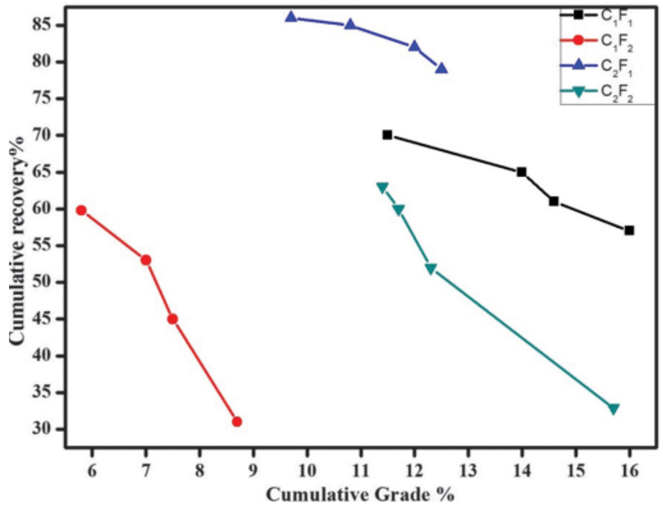

Fig.2 Different cumulative recovery and grade at four different reagent combination

It may be clearly observed that at slightly increased collector dosage i.e. C_2 (0.04kg/tonne) and lower frother dosage i.e. F_1 (0.02kg/tonne) both recovery and grade were maximum. The assay value of copper during C_1F_1 (C_1= 0.00kg/t, F_1= 0.02kg/t) condition is more as compared to C_2F_1 condition but the recovery is quite low. During collector-less condition (C_1=0.00kg/t, F_1=0.02kg/t), pine oil was used as frother which also exhibit slight collecting property. It may have introduced more hydrophobicity to the valuable mineral, therefore the grade of concentrate obtained is high. On further addition of collector (C_2=0.04kg/tonne) the recovery increased but the grade decreased slightly. In the finer size fraction of feed (–63micron), liberated copper content and liberated gangue content is more. So with increase in collector dosage from C_1–C_2, the collector may get adsorbed on the surface of valuable mineral as well as gangue mineral due to its finer sizes. The bubble may have attached to both copper and gangue particle and float them to the froth phase, leading to the increase in recovery but decrease in grade.

Thus, two values (minimum and maximum range) of each operating condition were considered for performing further tests.

Statistical design of experiments

Table 1 lists the design of experiment with coded variables and the results are presented in Table 2.

If all independent variables are assumed to be measurable, then the full factorial can be represented as

$Y = f(X_1, X_2, X_3 \ldots\ldots\ldots X_i)$

Where Y is the output and $X_{(1\text{-}i)}$ are the independent variables of action termed as factors (Aslan et al., 2009; Özgen et al., 2009).

The final equation for bulk is as follows:

$$Y_1 \text{ (Recovery)} = +72.38750+680.62500 * X_1-739.82955* X_2+0.001667* X_3 -10281.3 * X_1* X_2+2.94697* X_2* X_3 \quad \ldots (1)$$

$$Y_2 \text{(Grade)}= +19.14205-178.12500* X_1 -162.50000* X_2-0.021970* X_3+5593.75000* X_1* X_2 \quad \ldots (2)$$

Validation of model

Fig.3 shows the coefficient of determination (R^2), which implies that the equation is capable of representing the system under certain given experimental condition. Joglekar and May stated that for an acceptable fit of a model, the least value of R^2 should be 0.8 (Joglekar et al., 1987). R^2 was found to be 0.99 for recovery and 0.98 for grade .The R^2 values indicate that there is an acceptable relationship between actual and predicted values and therefore the models were significant.

Main and Interactional effect of variables

(a) For recovery

Effect of variable can be calculated as follows:

Effect of variable = $(\Sigma Y_+/ \Sigma n_+) - (\Sigma Y_- / \Sigma n_-)$

where 'Y_+ and Y_-' refers to the response at high level (+1) and lower level (–1) respectively, and 'n_+ and n_-' refers to the number of data points collected at high level and low level. [Table 3 (a)]

From Table 3 (a), it can be observed that the effect of X_1X_3 and $X_1X_2X_3$ is very less as compared to others. So these two trivial effects will be used as an estimate of error for analysis of variance (ANOVA). The five effects(X_1, X_2, X_3, X_1X_2 and X_2X_3) are most likely significant in a statistical sense.

(b) For grade: The effect of variable for grade is given in Table 3(b).

From the above Table 3 (b), it is analysed that the effect of X_2X_3, X_1X_3 and $X_1X_2X_3$ is very less as compared to others and so the effects of these three will be used as an estimate of error for analysis of variance (ANOVA). The remaining four effects (X_1, X_2, X_3 and X_1X_2) are most likely significant in a statistical sense.

Analysis of variance

Tables 4 and 5 show the ANOVA models of recovery and grade obtained by statistical design, which implies that flotation model is significant, as it is known that model is significant if p value is less than 0.05 ($p<0.05$).

In the Table 4, the individual influence of collector dosage, frother dosage and time were significant.

Recovery is denoted by the amount of solids reported to the froth. Frother acts on the air water interface and reduces the surface tension to stabilise the bubble-mineral attachment

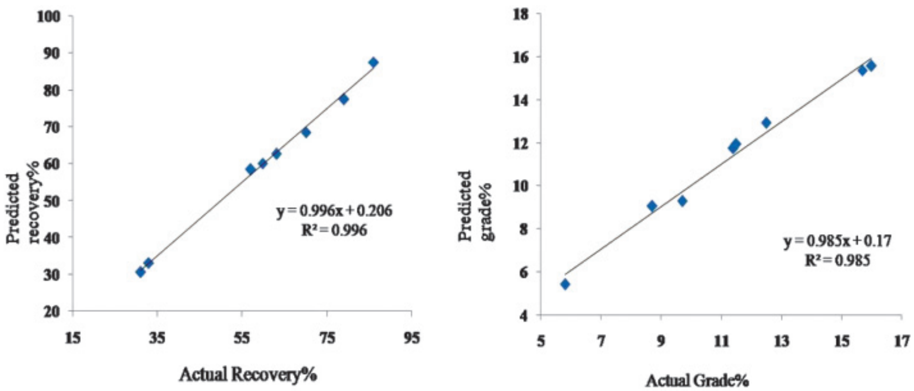

Fig.3 Relationship between actual and predicted value of recovery and grade of bulk

Table 1: Coded and actual level of independent variables used in the experimental design

Variable	Symbol	Unit	Coded variable level	
			-1	+1
Collector	X_1	kg/t	0	0.04
Frother	X_2	kg/t	0.02	0.06
Time	X_3	sec	15	180

Table 2: Full factorial design

Run	Coded level of variable			Actual level of variable			Recovery		grade	
	X_1	X_2	X_3	X_1	X_2	X_3	Actual	Predicted	Actual	Predicted
1	-1	-1	-1	0.00	0.02	15	57.0	58.5	16.0	15.60
2	+1	-1	-1	0.04	0.02	15	79.0	77.5	12.50	12.90
3	-1	+1	-1	0.00	0.06	15	31.0	30.7	8.70	9.10
4	+1	+1	-1	0.04	0.06	15	32.9	33.2	15.70	15.40
5	-1	-1	+1	0.00	0.02	180	70.0	68.5	11.50	11.90
6	+1	-1	+1	0.04	0.02	180	86.0	87.5	9.70	9.30
7	-1	+1	+1	0.00	0.06	180	59.8	60.1	5.80	5.40
8	+1	+1	+1	0.04	0.06	180	63.0	62.7	11.40	11.70

Table 3(a): Complex matrix showing calculated main and interaction effect on recovery

Standard	Main effect			Interaction effect				response
	X_1	X_2	X_3	X_1 X_2	X_2 X_3	X_1 X_3	X_1X_2 X_3	Y_1 (recovery)
1	-1	-1	-1	+1	+1	+1	-1	57.0
2	+1	-1	-1	-1	+1	-1	+1	79.0
3	-1	+1	-1	-1	-1	+1	+1	31.0
4	+1	+1	-1	+1	-1	-1	-1	32.9
5	-1	-1	+1	+1	-1	-1	+1	70.0
6	+1	-1	+1	-1	-1	+1	-1	86.0
7	-1	+1	+1	-1	+1	-1	-1	59.8
8	+1	+1	+1	+1	+1	+1	+1	63.0
Effect	10.775	-26.325	19.725	-8.225	9.725	-1.175	1.825	59.83

Table 3(b): Complex matrix showing calculated main and interaction effect on grade

Standard	Main effect			Interaction effect				response
	X_1	X_2	X_3	X_1 X_2	X_2 X_3	X_1 X_3	X_1 X_2 X_3	Y_2 (Grade)
1	-1	-1	-1	+1	+1	+1	-1	16.0
2	+1	-1	-1	-1	+1	-1	+1	12.5
3	-1	+1	-1	-1	-1	+1	+1	8.7
4	+1	+1	-1	+1	-1	-1	-1	15.7
5	-1	-1	+1	+1	-1	-1	+1	11.5
6	+1	-1	+1	-1	-1	+1	-1	9.7
7	-1	+1	+1	-1	+1	-1	-1	5.8
8	+1	+1	+1	+1	+1	+1	+1	11.4
Effect	1.825	-2.025	-3.625	4.475	0.025	0.075	-0.7751	11.41

Table 4: ANOVA for recovery of the concentrate

Source	Sum of squares	Df	Mean square	F value	p-value prob>f	
Model	2720.82	5	544.16	115.5	0.0086	Significant
X_1Collector	232.2	1	232.2	49.29	0.0197	Moderately significant
X_2Frother	1386.01	1	1386.01	294.19	0.0034	Very significant
X_3Time	778.15	1	778.15	165.17	0.0060	Very significant
X_1X_2	135.3	1	135.30	28.72	0.0331	Less significant
X_2X_3	189.15	1	189.15	40.15	0.0240	Less significant
Residual	9.42	2	4.71			
Cor total	2730.24	7				

Table 5: ANOVA for grade of the concentrate

Source	Sum of squares	Df	Mean square	F value	p-value prob>f	
Model	81.19	4	20.3	50.17	0.0044	Significant
X_1 Collector	6.66	1	6.66	16.46	0.0270	Moderately significant
X_2Frother	8.2	1	8.20	20.27	0.0205	Moderately significant
X_3Time	26.28	1	26.28	64.96	0.0040	Very significant
X_1 X_2	40.05	1	40.05	98.99	0.0022	Very significant
Residual	1.21	3	0.40			
Cor total	82.41	7				

and due to the buoyancy force they report to the froth phase. In this case, main effect of frother is more followed by time and collector. It may be due to the collecting property of frother (pine oil) which renders a higher frothing activity, resulting in probable gangue entrainment along with valuable mineral. Time shows also significant individual influence as time decides how much hydrophobic particles report to froth how quickly that ultimately decide the recovery. This is evidenced by the significance of the interactional effect between frother and time, collector and frother in that order on the recovery of bulk concentrate.

TABLE 6: INFLUENCE OF MAIN AND INTERACTION EFFECT OF PARAMETERS ON RECOVERY AND GRADE

Main effect	Interactional effect
a) Recovery- Frother > Time > Collector $X_2 > X_3 > X_1$	Frother*Time>Collector*Frother $X_2X_3 > X_1X_2$
b) Grade- Time>Frother>Collector $X_3 > X_2 > X_1$	Collector*Frother X_1X_2

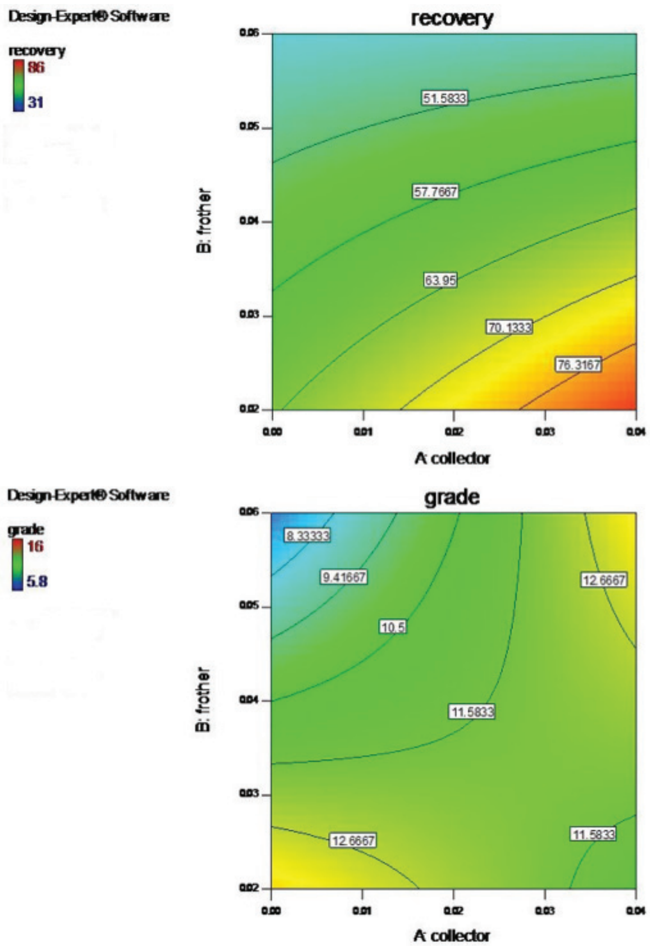

Fig.4 Contour plot of effect of the interactions (a) between frother and collector on recovery, (b) between collector and frother on grade

From the ANOVA Table 5, it can be seen that the individual effect of time on grade is more significant followed by frother and collector dosages. However, the interactional effect of frother-collector is significant. The grade is a factor of how rapidly and effectively the hydrophobicised values are transferred from pulp phase to froth phase. This higher rate of flotation of value can be ensured only when the conductive chemical environment is created by the collector and frother.

Table 6 shows the sequential influence of main and interaction effect of parameters on recovery and grade.

Influence of variables on responses

The relationship between variables and responses were studied and their effects on each other were analysed. Experimental results have been analysed through contour plots by software in Fig.4. From this graph, it may be observed that at minimum frother dosage and maximum collector dosage, recovery percentage is high (76.3%) while higher grade (12.6% Cu) can be observed at maximum collector and frother dosage as well as minimum collector and frother dosage. The grade (11.6% Cu) obtain at higher collector dosage and minimum frother dosage is acceptable.

Table 7 shows the best results obtained at optimum condition at which recovery and grade were adequetely higher.

Summary and conclusion

The full factorial methodology has been used to optimize the copper flotation process and find significant parameters. It also concluded that C_2F_1 (C_2 = 0.04kg/t, F_1 = 0.02kg/t) is the best condition for adequately higher responses (recovery and grade) in copper flotation. The Results of optimization concluded that recovery and grade could be 77.5% and 13% respectively when the value of collector, frother and time were 0.04kg/tonne, 0.02 kg/tonne and 15 sec respectively, which was approximately same as obtained from experiments. The predicted values obtained from the model equations were best fitted with observed values (R^2 value of 0.996 for recovery, R^2 value of 0.985 for grade). It would be prudent to analyze the behaviour of copper to effectively understand the flotation and arrive at a flotation strategy to maximise recovery and grade. From the optimization study it may be

TABLE 7: PROCESS OPTIMIZATION RESULT

Case	Target	Collector (kg/t)	Frother (kg/t)	Time (sec)	Recovery	Grade (Wt. %)	Desirability (Cu %)
Recovery variable grade	Maximum in range maximum	0.04	0.02	15	77.5	13	0.8

concluded that Rakha copper ore responds well to flotation and can be floated easily within a short interval of flotation time.

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